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Multifunctional Cement Composites Enhanced with Carbon Nanotube Thin Film Interfaces

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Abstract—Concrete and other cementitious composites used in civil infrastructure are prone to many forms of damage. For example, cracks could occur and propagate to cause catastrophic component or system-level failure. Therefore, the objective of this study was to nano-engineer a self-sensing cementitious composite that could detect and locate damage. Integration of multi-walled carbon nanotubes (MWNT) in the mortar mix design and during casting enhanced the electromechanical or sensing properties of the cementitious composites. Instead of directly dispersing MWNTs in the cement matrix, the cement-particle interface was modified using spray-coated MWNT-latex thin films deposited directly onto dry sand particles prior to mortar casting. This procedure preserved MWNT dispersions within the thin film architecture, used minimal amounts of MWNTs, was scalable, and provided dramatic sensing performance enhancements. The mechanical and electromechanical properties of mortar specimens were characterized using compressive cyclic load tests. Furthermore, an electrical resistance tomography (ERT) algorithm was used for mapping mortar specimens' spatial resistance distributions. Since their resistance was pre-calibrated to strain, localized changes in calculated resistance indicated damage. Spatial damage detection was validated by drilling holes in various locations in cementitious composite plates and then by mapping resistance distributions using ERT.

Index Terms—carbon nanotube, cement composite, damage detection, electrical resistance tomography, multifunctional material, thin film, structural health monitoring.

I. INTRODUCTION

Concrete is one of the most commonly used materials for civil infrastructure systems worldwide, and their applications extend beyond bridges, high-rises, pavements, foundations, and dams. For instance, ~343,000 of the ~607,000 bridges in the U.S. are made of pre-stressed or cast-in-place concrete [1]. The U.S. interstate highway network, which is over 72,000 km (45,000 miles) long, is comprised of ~60% concrete [2]. In addition, the demand and use of concrete has been increasing steadily [3]; this trend is comparable to other places in the world, such as China and India, whom remain the two largest

producers of cement [4]. In general, concrete's intrinsic properties, versatility, and cost effectiveness make them ideally suited as a civil infrastructure construction material.

Unfortunately, concrete and cementitious composites are susceptible to damage during service. Damage can stem from unanticipated extreme loads, long-term degradation, fatigue, and environmental effects, among others. In fact, cracks can form in response to tension, cyclic loads (fatigue), and freeze-thaw cycles [5]. Specifically, freeze-thaw cycles cause water in the pores of the cementitious matrix to expand (when frozen) and create new or widen existing cracks. Such effects exacerbate damage by allowing water, air, and other chemicals to seep beneath the surface, degrade the passivation layer, and induce corrosion of steel reinforcement bars [5, 6]. Corrosion byproducts (or rust), like ice, take up more volume and can cause additional cracking or spalling of the concrete cover. Over time, deterioration and damage can propagate to cause catastrophic structural collapse, as was the case for the Interstate-70 Lake View Drive Bridge failure in 2005 (Pennsylvania, U.S.) [7].

Thus, detecting the onset and monitoring the progression of damage are critical for facilitating necessary maintenance and for preventing catastrophic failure. For the specific case of cementitious composite structures (e.g., bridges and concrete pavements), the approaches for detecting damage can be classified as either destructive or nondestructive. A common destructive test is core sampling, in which a core sample of a specified diameter and depth is drilled and extracted from an existing concrete structure [8]. The specimen is then subjected to laboratory tests for characterizing its mechanical properties, as well as to infer any possible deterioration occurring in the structure. While this approach provides invaluable information about the concrete structure, it has the drawback of altering the structure destructively and potentially creating a "weak-spot" in which damage can initiate and occur in the future.

In contrast, nondestructive testing (NDT) does not alter the structure but instead utilizes technologies or methods that extract structural response data that are correlated to damage [9]. Visual inspection is usually the most common evaluation method employed for monitoring concrete structures. Despite its widespread usage, visual inspection remains to be time-, cost-, and labor-intensive and can be subjective. Taking digital images of suspected damaged regions and the application of image processing techniques enable quantification of damage, but the technique is limited to surface features [10]. On the other hand, acoustic emission (AE) is another NDT technique

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that uses ultrasonic transducers to characterize changes in the acoustic properties of the structure due to damage (*e.g.*, cracks) [11], which is somewhat related to impact-echo and chain dragging methods that rely on measuring changes in wave propagation properties in the material due to damage. The drawbacks of AE include its inability to detect pre-existing damage and its susceptibility to ambient noise [12].

Over the last few decades, a different approach for damage detection has been proposed and is based on designing a new generation of multifunctional cementitious composites. In general, the addition of conductive additives to the cement matrix has enabled multifunctional cementitious composites to bear loads while possessing self-sensing properties [13]. As early as 1995, Chen and Chung [14] demonstrated that the electrical resistance of concrete mortars with carbon fibers dispersed in the mortar matrix changed in response to cyclic loading. While resistance change was reversible during cyclic loading, cracks or damage caused irreversible changes in the “smart” concrete’s resistance. Later improvements involved using ozone-treated carbon fibers, which led to improvements in the sensing response’s sensitivity and repeatability. The incorporation of steel and carbon fibers together in concrete beams was also studied; not only was this combination successful in altering the concrete’s mechanical properties (*i.e.*, flexural strength and toughness), but its electrical resistance was also sensitive to crack width [15]. However, it should be mentioned that a limitation of using these fibers as conductive additives is to make sure that they are evenly distributed within the composite. Poor dispersion or agglomerations would diminish material properties. In addition, existing construction practices need to be modified for casting fiber-reinforced cementitious composites, which may be a deterrent for practical implementations.

More recently and with the advent of nanotechnology, the design of multifunctional or smart cementitious composites focused on leveraging the unique properties of conductive nanomaterials such as carbon nanotubes (CNT) [16–19]. For example, Yu and Kwon [20] fabricated cement paste specimens and investigated two methods for dispersing CNTs, namely using acid treatment to form functionalized carboxylic groups on CNT surfaces and using non-covalent suspension in surfactants. In fact, different groups used various surfactants, including sodium dodecyl sulfate [20, 21], sodium dodecyl benzene sulfonate [21, 22], and poly(sodium 4-styrene sulfonate) [16, 23], to name a few. In fact, Han *et al.* [18] showed that CNT-based cementitious pavements can be used for traffic monitoring. Besides CNTs, other nanomaterial additives were also investigated, such as carbon nanofibers [24], carbon black [17], and graphite [25]. While these studies demonstrated promise for achieving next-generation, self-sensing, cement composites, it remains challenging to scale up due to dispersion issues, specialized casting procedures, and cost.

Therefore, the objective of this study was to develop a multifunctional cementitious composite that could be used as a structural or load-bearing material while providing the added functionality of sensing strain and damage. Instead of

Fig. 1. The ERT and EIT algorithms seek to reconstruct the spatial resistivity (or conductivity) distribution of a conductive body using only boundary electrode current inputs/outputs and corresponding sets of boundary voltage measurements. A two-dimensional finite element model of the body was generated using quadrilateral elements with bi-linear basis functions.

dispersing CNTs in the cement matrix, this study focused on modifying the cement-sand interface of mortar specimens with CNT-based thin films [26]. In short, an airbrush was employed for spraying electrically conductive CNT-latex thin films onto dry sand. The film-coated sand was then used for casting mortar specimens. Upon casting, mortar specimens were subjected to compressive and cyclic load tests for characterizing their mechanical and sensing properties. Once the mortar’s electrical properties were calibrated to applied strains, an electrical resistance tomography (ERT) algorithm was implemented and used for estimating the specimen’s spatial distribution of electrical resistance. In doing so, direct spatial sensing and damage identification could be accomplished, since localized changes in electrical resistance would suggest the presence of cracks, spalling, or other types of damage.

This paper begins with a description of the ERT theoretical formulation. Second, the experimental details, namely the mix design, CNT thin film fabrication method, casting procedures, and testing protocols, are described. Then, the paper continues with a discussion of the mechanical and electromechanical test results. Spatial damage detection using these multifunctional cementitious composites coupled with the ERT algorithm was also validated. The paper ends with a brief conclusion and discussion of future research directions.

II. ELECTRICAL RESISTANCE TOMOGRAPHY BACKGROUND

Electrical resistance tomography, which is a subset of the broader domain of electrical impedance tomography (EIT), is a well-known soft-field tomography technique [27], and its development and applications can be traced back to the biomedical domain [28]. ERT and EIT seek to reconstruct the spatial distribution of resistance and impedance, respectively, of a conductive body using only applied electrical input and measured output signals along the body’s boundaries. In that regard, ERT and EIT use experimental data of input current and boundary voltage measurements for solving the inverse problem to reconstruct the spatial conductivity map of the body of interest (Fig. 1) [27]. The main difference between the two techniques is that ERT employs direct current (DC) electrical excitations for reconstructing spatial resistance distributions, whereas EIT benefits from the use of alternating

current (AC) excitations to yield more information about the conductive body's electrical impedance at various frequencies.

To understand the ERT and EIT spatial conductivity mapping inverse problem, one must first consider the "forward problem." Using the 2D Laplace's equation,

$$\nabla \cdot [\sigma(x, y) \nabla v(x, y)] = 0 \quad (1)$$

one can calculate the body's boundary potential or voltage distribution (v) given a known electrical current source/sink and the body's conductivity distribution (σ) represented by a Cartesian coordinate system. Equation (1) refers to the specific case in which electrical current is neither supplied nor generated within the 2D body, Ω . No discrete electrodes are defined in this continuum model. Instead, electrical current is defined as a continuous current function along the boundary, $\partial\Omega$ [29].

Given the complexity of the ERT and EIT problem and the inability of applying a continuous boundary current function to Ω , the forward problem is often solved using a discretized weak formulation in conjunction with the finite element method (FEM) (Fig. 1). Equation (2) shows the weak form of Laplace's equation:

$$\iint_{\Omega} \sigma \nabla \phi \nabla v \, dx \, dy = 0 \quad (2)$$

where ϕ is the shape function of voltage at the electrodes. FEM and discretization allows the incorporation of discrete boundary electrodes, which is more practical than applying a continuous boundary current function. In addition, each FEM element assumes a constant value of conductivity or resistivity [30, 31]. In this study, four-node quadrilateral elements were implemented.

While the forward problem can be solved using (2), ERT and EIT involve solving the "inverse problem." Given a physical Ω with a set of discrete boundary electrodes, one can simply apply a DC current at one electrode, set another electrode as ground, and measure the potential drops or voltage differences at remaining pairs of boundary electrodes (v_{exp}), as shown in Fig. 1. Here, $\sigma(x, y)$ is unknown. Thus, solving the inverse problem begins by assuming a conductivity distribution so that the forward problem can be executed to calculate the predicated boundary voltages (v_{num}). In this work, a Gauss-Newton iterative algorithm was implemented for updating the body's absolute conductivity or resistivity distribution until the error ratio (e) of the norm of the differences between experimental and predicted voltages and the norm of experimentally measured boundary voltages was below a certain threshold as indicated in (3):

$$e = \frac{\|v_{exp} - v_{num}\|^2}{\|v_{exp}\|^2} \leq 0.05\% \quad (3)$$

It should be mentioned that the nature of ERT reconstruction is an ill-posed inverse problem, which necessitates the inclusion of some prior regularization information in the reconstruction algorithm [27]. In this work, a Tikhonov regularization scheme was employed.

Fig. 2. A digital microscope image of a sand particle coated with a multi-walled carbon nanotube-latex thin film is shown. Film-enhanced sand was used as is for mortar specimen casting.

III. EXPERIMENTAL DETAILS

An experimental program was devised for characterizing the mechanical and sensing properties of multifunctional cementitious composites. As mentioned in Section I, the cement-sand interface of the cementitious composite was enhanced by directly depositing CNT-based thin films onto sand particles prior to using them for mortar casting (Fig. 2). Although other studies demonstrated some success by dispersing nanomaterials in cement matrices [19], the objective of this study was to explore a new and more efficient method of incorporating carbon nanotubes and for enhancing electromechanical performance. Section III.A starts by explaining the mortar mix design and casting procedures. Second, Section III.B discusses the procedure for preparing the CNT ink solution and for spraying films onto sand. Finally, Section III.C describes the load test setup, while Section III.D explains how spatial damage detection validation using the ERT algorithm was performed.

A. Mix Design and Casting

Mortar specimens were cast following the procedure developed in a previous study [32], which was a combination of the Federal Aviation Administration (FAA) guidelines for runway concrete pavement [33] and ASTM C109 [34] for mortar specimen strength testing. The cementitious material used was a mixture of Type I/II Portland cement and 25 wt.% (of total cementitious material) Type F ground granulated blast furnace slag (GGBFS). Specimens were cast using a 2.75:1 sand-to-cement ratio, as stated in ASTM C109 [34]. The sand used was crushed granite with a density of 2.71 g/cm³ and a minimum particle size of 0.149 mm. Prior to its use and/or the deposition of the CNT-based thin film (Fig. 2), sand was dried in a vacuum oven operated at 80 °C for 24 h. A water-to-cement ratio of 0.52 was used, along with 2.9 mL/kg of Advacast superplasticizer (SPL), for reducing water content and increasing mix workability.

Casting the mortar and plate specimens began with mixing the various constituents using a Hobart N-50 mixer. First, the cementitious material, water, and Advacast SPL were mixed at a low speed of 60 rpm for 30 s. While the mixer was still

Fig. 3. A saturated $5 \times 5 \times 5 \text{ cm}^3$ mortar specimen with embedded copper mesh electrodes is shown.

operated at 60 rpm, sand (*i.e.*, either pristine or coated with the CNT-based thin film) was gradually added. Upon doing so, the mixer was set to medium-speed (124 rpm) for 1 min, so as to obtain an even mixture. Then, the mixer was turned off, and any material on the walls of the bowl was scraped off. Finally, the mixture was set aside for 15 s before being poured into individual molds.

Two different types of molds were used for casting mortar specimens in this study. The first type was $5 \times 5 \times 5 \text{ cm}^3$ ($2 \times 2 \times 2 \text{ in}^3$) cubic molds. These molds were oiled with WD-40 as a releasing agent, and the seams were sealed with petrolatum. In addition to subjecting these cubic specimens to mechanical tests, the sensing characterization tests required that their electrical properties be measured during applied strains and stresses. For this reason, $4.5 \times 6.5 \text{ cm}^2$ ($1.75 \times 2.25 \text{ in}^2$) copper mesh electrodes were cut and inserted at opposite ends in certain molds (*i.e.*, those designated for casting specimens for electromechanical characterization). Electrode placement was done so that the mesh covered the entire specimen's cross-section while having a small portion extended outwards for measurement purposes. Once the electrodes were in place, the mix was scooped into each mold, filling them halfway. Each mold was then tamped for 10 s before being filled completely and tamped again for another 10 s. After tamping, the molds were sealed with plastic wrap and stored at room temperature in a sealed container for 24 h. Finally, the specimens were demolded and placed in a limewater solution to cure for 28 days prior to testing. A saturated mortar specimen with embedded electrodes is shown in Fig. 3. For this study, mortar cube specimens using pristine sand and film-coated sand were cast for testing purposes and for comparison.

For the second sample set, customized $25 \times 12.5 \times 1 \text{ cm}^3$ poly(vinyl chloride) (PVC) molds were used for casting plate specimens for spatial damage detection validation tests. These plate molds were also oiled with WD-40 and sealed with petrolatum before use. Because these specimens were to be used for ERT testing, 24 copper tape electrodes were arranged to form a 6×6 square pattern similar to Fig. 1. The electrodes,

Fig. 4. The scanning electron microscope image shows the morphology of the MWNT-latex thin film, which consists of dispersed nanotubes dispersed and intermixed with latex spherical particles $\sim 100 \text{ nm}$ in size.

which were 5 mm wide and spaced 5 mm apart, were arranged using a balsa wood form that created a $6.5 \times 6.5 \text{ cm}^2$ square layout. Once the forms and molds were ready, the mixture was poured into each mold and tamped for 20 s using a trowel. The entire mold was then gently vibrated by hand and then wrapped in plastic wrap. The curing procedure was the same as that of the cubic specimens. A total of three $7 \times 7 \times 1 \text{ cm}^3$ specimens was obtained from each plate. Only film-coated mortar plates were casted for the damage detection study.

B. Nanocomposite Fabrication and Sand Coating

As stated earlier, the sand was coated with a multi-walled carbon nanotube (MWNT)-based thin film prior to using them for casting. In this study, the film was sprayed and deposited onto dry sand using an airbrush, as was described by Mortensen *et al.* [35]. Prior to spraying films, an MWNT solution was prepared. First, MWNTs were dispersed in 2 wt.% poly(sodium 4-styrenesulfonate) (PSS) solution with a small amount of N-methyl-2-pyrrolidinone (NMP). After achieving adequate suspension by bath and high-energy tip sonication [36], a latex solution was added to create the MWNT-latex ink, which was used as is for spraying using a Paasche airbrush. As was discussed in Mortensen *et al.* [35], both the PSS and latex facilitated MWNT suspension and the deposition of films characterized by a percolated morphology and high homogeneity, as can be seen from the scanning electron microscope (SEM) image in figure 4.

The procedure for coating sand with the MWNT-latex film began with spreading sand on a clean flat surface. Then, the Paasche airbrush and MWNT-latex ink solution was employed for spraying a thin film onto the sand surface. The airbrush was positioned 30 cm above and perpendicular to the sand surface, while film was deposited using a sweeping motion. After applying one coating, sand was mixed thoroughly using

Fig. 5. The ERT DAQ interrogated a center hole-damaged mortar plate specimen (that incorporated MWNT-latex thin films airbrushed onto sand) so as to obtain the necessary boundary voltage measurements for reconstructing its spatial resistivity map.

a plastic spatula to expose any untreated sand. Airbrushing was repeated two more times. Finally, the film-coated sand was left to air-dry for 1 h, followed by drying at 50 °C for 30 min in a vacuum oven. It should be mentioned that, while uniform films can be deposited onto flat surfaces, it is likely that the films on sand exhibited non-uniform thicknesses (*i.e.*, due to the orientation, angularity, and gradation of sand particles). However, it is hypothesized that this would not affect the electrical and electromechanical properties of the bulk cementitious composite.

C. Load Testing

An MTS load frame equipped with a 407 controller was employed for testing mortar specimens and for characterizing their mechanical and electromechanical properties. The cubic mortar specimens (Section III.A) were used for two types of tests. First, specimens without embedded electrodes were subjected to monotonic, uniaxial, compressive loading until material failure so that their mechanical properties could be characterized. The displacement-controlled test was conducted using a ramp rate of 1.905 mm/min. Eight pristine and seven thin film-enhanced mortar specimens were tested for this case.

The second set of tests was for characterizing the electromechanical response of mortar specimens. Thus, cubic specimens with embedded copper mesh electrodes were used. The specimens were initially preloaded to 5,000 N, before the load frame executed a 0.1 Hz compressive cyclic load pattern to a peak strain of -0.45%; they were subjected to a minimum of five load cycles. The load frame's load and displacement data were recorded using a customized *LabVIEW* client (sampling rate: 55 Hz). In addition, its DC resistance was also recorded simultaneously using an Agilent 34401A digital multimeter (DMM) recording at a sampling rate of 2.8 Hz. It should be mentioned that, prior to each test, ~20 s of resistance data was recorded first. This data was used for quantifying the specimen's nominal resistance, as well as for identifying any possible drift in its electrical response.

It should be mentioned that all of the specimens tested in

this study were dried at 50 °C for 6 h in a vacuum oven immediately prior to testing. It was common to conduct multiple tests on the same specimen. However, the time between each test was minimized so as to reduce experimental error induced by possible moisture changes that could occur in each specimen. In total, six pristine and six film-enhanced mortar specimens were subjected to electromechanical tests.

D. Spatial Damage Detection Validation

A testing protocol was established so as to validate the use of ERT for detecting the severity and location of damage occurring in mortar specimens. For this test, only plate specimens (that have been enhanced with MWNT-latex films airbrushed onto dry sand particles) with the 6×6 copper mesh electrodes were used (Fig. 5). A specialized data acquisition (DAQ) system was employed so as to obtain the dataset for mapping the distribution of electrical resistance in the mortar specimens. The ERT DAQ included a Keithley 6221 current source, which supplied DC on the order of 1 mA to a specific boundary electrode and with another electrode set as ground. It should be noted that the DC magnitude was set to span the voltage measurement range of the DAQ, thereby maximizing signal-to-noise ratio. On the other hand, an Agilent 34980A multifunction switch was employed for measuring boundary electrode voltages (using a built-in DMM) and for directing current input/output to appropriate electrode pairs. The ERT DAQ was controlled using a customized *MATLAB* program. In this study, DC was injected across electrodes on opposite edges of the square mortar specimen, while voltage was measured across adjacent boundary electrodes (Fig. 5); a similar approach was used by Loyola *et al.* [37].

Two sets of tests were conducted for validating spatial damage detection using the film-enhanced mortar specimens coupled with the ERT algorithm. In either case, testing began with obtaining a baseline or undamaged spatial resistivity map using the boundary voltage dataset collected by the ERT DAQ. Then, the first test involved drilling a 6.35 mm (0.25 in)-diameter hole through the center of the plate specimen for simulating damage. The DAQ was then used to interrogate the mortar specimen and to obtain the corresponding boundary voltage dataset. On the other hand, the second test used the same drill but considered inflicting different levels of damage at different locations. A hole was drilled halfway through the specimen near its bottom-left corner, and this simulated minor damage in the structure. The drill was then used again, at the same location, to drill through the plate to create more severe damage. This process was repeated near the top-right corner as well. After each drilling action, the ERT DAQ was employed to interrogate and obtain the boundary voltage measurements so that the spatial resistivity maps corresponding to each damage state could be reconstructed.

IV. RESULTS AND DISCUSSION

As mentioned in Section III, mortar specimens were cast and subjected to both mechanical and sensing characterization tests. This section discusses the results obtained and compares the results of pristine mortar versus specimens casted using

Table I. Comparison of mortar specimens' Young's modulus

Pristine mortar	E [ksi]	E [GPa]	MWNT mortar	E [ksi]	E [GPa]
#M-P1	2,060	14.2	#M-M1	800	5.52
#M-P2	1,980	13.7	#M-M2	905	6.24
#M-P3	2,210	15.2	#M-M3	820	5.65
#M-P4	2,280	15.7	#M-M4	750	5.17
#M-P5	1,890	13.0	#M-M5	950	6.55
#M-P6	1,730	11.9	#M-M6	1020	7.03
#M-P7	1,990	13.7	#M-M7	876	6.04
#M-P8	2,100	14.5			
Average	2,030	14.0	Average	874	6.03
Standard deviation	175	1.2	Standard deviation	92.7	0.64

Table II. Comparison of mortar specimens' compressive strength

Pristine mortar	f_c' [psi]	MWNT mortar	f_c' [psi]
#M-P1	5,290	#M-M1	3,220
#M-P2	4,820	#M-M2	3,270
# M-P3	5,160	#M-M3	3,740
# M-P4	4,830	#M-M4	2,900
# M-P5	4,630	#M-M5	3,300
# M-P6	4,110	#M-M6	3,560
# M-P7	4,550	#M-M7	3,190
# M-P8	4,720		
Average	4,760	Average	3,310
Standard deviation	370	Standard deviation	270

MWNT-latex thin film-coated sand.

A. Mechanical Properties

Tables I and II summarize the pristine and MWNT-latex thin film-enhanced mortar specimens' mechanical properties. The first '#M' denotes mechanical tests, and the second character denotes the type of specimen, namely 'P' for pristine and 'M' for MWNT. The data presented in Table I was obtained from monotonic, uniaxial, compressive tests performed using $5 \times 5 \times 5$ cm³ cubic mortar specimens, as was discussed in Section III.A. It should be mentioned that these tests loaded the mortar specimens until failure. However, Young's modulus (E) was derived from the slope of the linear least-squares best-fit line applied to the initial linear portion of the stress-strain raw data. The average Young's modulus of the pristine and MWNT-latex thin film-enhanced mortar specimens was 14.0 ± 1.2 and 6.03 ± 0.64 GPa, respectively (Table I). The peak compressive strength (f_c') was taken as the peak value of the stress-strain raw data (Table II).

From the results presented in Tables I and II, it was clear that the MWNT-latex films affected the stiffness and strength

Table III. Comparison of mortar specimen nominal electrical properties

Pristine mortar	Average resistivity [k Ω -m]	MWNT mortar	Average resistivity [k Ω -m]
#E-P1	22.9	#E-M1	<u>38.5</u>
#E-P2	21.9	#E-M2	2.8
#E-P3	27.8	#E-M3	2.38
#E-P4	22.5	#E-M4	2.12
#E-P5	<u>2.73</u>	#E-M5	1.82
#E-P6	<u>2.77</u>	#E-M6	1.56
Average	23.8	Average	2.14
Standard deviation	2.7	Standard deviation	0.48

of pristine mortar. It is known that the strength and stiffness of cementitious composites are derived from the cement-particle interactions. It was possible that MWNT-latex films coated on sand prevented those same types of chemical bonds to form, hence decreasing Young's modulus and compressive strength of the bulk mortar specimen. Another reason may be because of particle smoothing due to the MWNT-latex coating, which reduced the ability of particles to interlock and resist loads.

B. Nominal Electrical Properties

In addition to the specimens' mechanical properties, their nominal or unstrained electrical properties were also quantified. As was discussed in Section III.A, DC resistances of six pristine and six MWNT-latex thin film-enhanced mortar cubic specimens were measured prior to electromechanical testing. For each specimen, ~20 s of resistance data (sampling rate: 2.8 Hz) was collected. The average DC resistance for each specimen was computed and presented in Table III. In Table III, '#E' denotes nominal electrical characterization tests, and 'P' and 'M' refer to the two different types of sample sets tested in this work.

One can see from the results shown in Table III that the average resistivity values of mortar specimens without and with MWNT-latex thin films were drastically different. Outliers were present (*i.e.*, indicated as underlined values in Table III), and they could be a result of improper drying or electrode corrosion during curing. Without considering these potential outliers in the nominal resistivity data, it was found that the average resistivity was 23.8 ± 2.7 and 2.14 ± 0.48 k Ω -m for the pristine and film-enhanced mortar, respectively. The resistivity difference between the two sets was more than one order of magnitude, which is expected given the high electrical conductivity of carbon nanotubes and of the MWNT-latex thin films [35]. It should be mentioned that a few of the specimens tested exhibited significant nominal resistance drifts, and those results were not included in this study.

C. Sensing Response Characterization

The electromechanical properties of cubic mortar specimens were characterized by conducting compressive cyclic tests, as

Fig. 6. (a) The resistivity time history response of mortar specimen #S-M1 (*i.e.*, with MWNT-latex thin film) is overlaid with the applied strain time history. The results clearly show that the specimen's electromechanical properties are sensitive to applied strains. (b) Upon plotting the normalized change in resistance with respect to applied strains, it was found that the electromechanical response was bilinear.

Fig. 7. (a) The resistivity time history response of mortar specimen #S-M6 (*i.e.*, with MWNT-latex thin film) is overlaid with the applied strain time history. The results clearly show that the specimen's electromechanical properties are sensitive to applied strains. (b) Upon plotting the normalized change in resistance with respect to applied strains, it was found that the electromechanical response was bilinear.

was discussed in Section III.A. During these tests, the specimen's DC resistance was recorded, along with its force-deformation response. It should be noted that the applied peak compressive strain was approximately -0.45%. This peak strain was selected to ensure that the mortar specimens remained linear-elastic and undamaged.

Figs. 6a and 7a plot two representative results of the electromechanical tests; the plots show the mortar specimen's resistivity time histories overlaid with that of the applied strain patterns. Both sets of these results were for mortar specimens that incorporated MWNT-latex thin films. As expected, the test results showed that the mortar specimens' electrical properties were sensitive to applied strains. In addition, resistivity decreased in tandem with decreasing strains (*i.e.*, with greater magnitudes of applied compressive strains). It should be mentioned that the pristine mortar specimens also exhibited similar resistivity changes with respect to applied compressive loading. It can also be seen from Figs. 6a and 7a that resistivity change was synchronized with that of the

applied strains, and the specimen's sensitivity to strain was stable and repeatable.

While no significant nominal resistivity drift was observed during loading, resistivity drift, potentially due to polarization, was present during the initial 20 s of resistance measurements. For most cases, the nominal resistivity drift plateaued quickly, and tests were conducted after such drifts disappeared. In fact, an earlier studied showed that the nominal resistivity drift of cement paste specimens with ultra-low concentrations of MWNTs would eventually plateau after repeated cyclic loading [38]. It was suspected that resistivity drift occurred because of electric charging or polarization. While the cement paste specimens tested by Wong *et al.* [38] were different than those presented in this study, polarization could still occur. It is worth noting that other researchers also observed similar charging effects, and its effect could be counteracted by applying AC excitations, using techniques such as applying a linear fit to the data [21], or by pre-charging the cementitious composite [39], just to name a few. Nevertheless, one can see

Table IV. Strain sensitivities of mortar specimens enhanced with MWNT-latex thin films

Low-strain gage factor							
Specimen	#S-M1	#S-M2	#S-M3	#S-M4	#S-M5	#S-M6	Average
$S_{low-strain}$	273	136	178	292	253	242	229±60
High-strain gage factor							
$S_{high-strain}$	56.5	87.2	59.6	62.9	61.0	63.9	65±11
Entire dataset gage factor							
$S_{complete}$	114	128	111	144	131	119	125±12

Table V. Strain sensitivities of pristine mortar specimens

Specimen	#S-P1	#S-P2	#S-P3	#S-P4	#S-P5	#S-P6	Average
$S_{complete}$	77.7	85.0	75.6	58.8	40.3	30.2	61±22

from Fig. 7a that the specimen's nominal resistivity was just beginning to plateau during the first few loading cycles.

For both of the aforementioned specimens, the same sets of data were converted such that normalized change in resistivity was plotted with respect to applied strains, as can be seen in Figs. 6b and 7b. Both Figs. 6b and 7b show that the electromechanical response of the mortar specimens (*i.e.*, for ones enhanced with the MWNT-latex thin film interface) exhibited a polynomial relationship with respect to applied strains but was approximated as bi-linear for the purposes of quantifying strain sensitivities. This observation was true and applicable for all of the specimens tested in this study. In addition, a linear least-squares regression line was fitted to each of the two linear portions for each dataset so as to determine the strain sensitivity (S) or gage factor, which can be calculated by the following:

$$S = \frac{\Delta\rho/\rho_0}{\Delta\varepsilon} \quad (5)$$

where ρ_0 is the nominal or unstrained resistivity, and $\Delta\rho$ is the change in specimen resistivity corresponding to the change in applied strains ($\Delta\varepsilon$).

Equation (5) was employed for estimating the low-strain and high-strain strain sensitivities for all the electromechanical test results. Since each mortar specimen was subjected to multiple loading cycles, low-strain S and high-strain S were calculated for each cycle, and the average strain sensitivities were computed. The results are summarized in Table IV. Here, the specimens are denoted as ‘#S’ for sensing characterization; the notations for ‘M’ and ‘P’ follow the same definitions in Section IV.A.

It should be mentioned that the pristine mortar specimens did not exhibit bi-linear strain sensitivity. As a result, each pristine mortar specimen was characterized by an average strain sensitivity value, as opposed to being separated to that of low-strain and high-strain; those results are tabulated in

Table V. For comparison purposes, Table IV also includes the strain sensitivities computed using the entire dataset ($S_{complete}$); to be specific, a linear least-squares regression line was fitted to the entire data for each cycle of loading; while this was not accurate, this calculation was performed so that a direct comparison can be made between the film-enhanced and pristine mortar sample sets.

One can observe from the results shown in Table IV that the average low-strain gage factor ($S_{low-strain}$) was very impressive at 229 ± 60 . This sensitivity was valid for applied strains between 0% and ~0.2%. It is hypothesized that such high strain sensitivities were obtained, because the embedded thin film-coated sand in unstrained mortar specimens was near or at the electrical percolation threshold. Any minor compressive load would cause a dramatic change in percolation properties, which would result in a large gage factor [40]. Applications of greater loads should cause strain sensitivity to decrease thereafter (*i.e.*, after exceeding the percolation threshold).

In fact, after the low-strain regime, the average $S_{high-strain}$ was 65 ± 11 . From representative plots shown in Fig. 6b and 7b, it is clear that bi-linear sensitivity was a good approximation and that within each low- or high-strain regime, linearity was strong. It is hypothesized that this bi-linear response was observed because, during initial loading, slight changes in orientation of MWNT-latex thin film-coated sand particles could occur and make contact with another particle, thereby changing the bulk specimen's electrical properties in a dramatic fashion. Localized high stresses acting on the film surface could also explain why such drastic changes in electrical properties were observed, especially since these MWNT-latex thin films are piezoresistive [35]. After this point, it is hypothesized that strain sensitivity is dominated by the cementitious matrix, although electrical conductivity of the bulk mortar is still enhanced with the presence of the MWNT-latex thin film.

In contrast, the pristine mortar specimens did not exhibit bi-linear strain sensitivity, and its average strain sensitivity was much lower, approximately 61 ± 22 . As compared to the MWNT-latex thin film-enhanced mortar, the pristine mortar possessed gage factors similar to that of $S_{high-strain}$ for the MWNT-based specimens. The comparable strain sensitivity makes sense, since it was hypothesized that the cementitious matrix was contributing to most of the electromechanical response observed. However, it should be mentioned that the standard deviation for the pristine mortar was 22, which is twice that of the MWNT-based specimens (*i.e.*, 11). In fact, the signal-to-noise ratio (SNR) and sensing resolution of the pristine mortar specimens was fairly low. Thus, despite comparable high-strain gage factors, the advantages offered by modifying the cement-sand particle interface with MWNT-latex thin films were improved SNR, sensing resolution, higher conductivity, and repeatability of measurements.

D. Spatial Damage Detection

Once the electromechanical properties of mortar specimens were characterized, the final phase of this study was to validate spatial damage detection. Plate specimens with 24

Fig. 8. An MWNT-based mortar plate was instrumented with 24 boundary electrodes, and a 6.35 mm-diameter hole was drilled through the center. The ERT algorithm was executed using the undamaged baseline and damaged boundary voltage datasets, and the resulting relative change in spatial resistivity distribution clearly shows the location and severity of damage near the center of the specimen.

embedded copper tape or mesh electrodes were cast, following steps outlined in Section III.A. It should be mentioned that only MWNT-based mortar plates were investigated, because preliminary tests showed that electrical current could not be effectively propagated through pristine mortar specimens. Because of this, spatial resistivity mapping of pristine mortar using the ERT algorithm was not possible.

Therefore, using the MWNT-latex thin film-enhanced plate specimens, two sets of damage detection tests were conducted, as was described in Section III.D. Fig. 8 shows the spatial resistivity map (after executing the ERT algorithm using the undamaged and damaged boundary voltage datasets) of a $7 \times 7 \times 1$ cm³ plate after a 6.35 mm-diameter hole was drilled through the center of the specimen (Fig. 5). It should also be mentioned that the resistivity map shown in Fig. 8 is the change in spatial resistivity distribution of the damaged ERT map relative to its undamaged baseline. Regardless, one can see that Fig. 8 clearly identifies an increase in resistivity corresponding to the location of the actual drilled hole. The estimated size of damage is slightly larger than the actual damage, and this is most likely due to the inherent resolution of the ERT algorithm, which is governed in part by electrode size and spacing. In addition, it is known that sudden jumps in conductivity (as was the case for the drilled hole) cannot be resolved precisely using ERT and is typically estimated with a gradual transition zone (due in part to regularization) as shown in Fig. 8. Nevertheless, Fig. 8 validates the ERT algorithm and its spatial damage detection capabilities.

According to Section III.D, an additional set of tests using these plates with 24 boundary electrodes, were also conducted. Instead of detecting binary cases (*i.e.*, whether or not damage exists in the structure), this test sought to validate the performance of the MWNT-based mortar and ERT algorithm for monitoring and detecting the severity and location of progressive damage. First, a partial hole (~50% through the depth of the specimen) was drilled near the lower-left corner of the plate (Fig. 9a). The ERT algorithm was used for reconstructing the change in spatial resistivity, which

Fig. 9. An MWNT-based mortar plate was subjected to spatial damage detection validation tests. (a) A partial hole was drilled near the bottom-left corner, and (b) the corresponding spatial resistivity map relative to its undamaged baseline is shown. (c) The hole is drilled completely through the plate, and (d) the spatial resistivity map relative to the baseline shows damage accumulation near the bottom-left, which is expected. (e) Another partial hole was created near the top-right corner, and (f) the spatial resistivity map (relative to the baseline) successfully detects damage occurring at the correct location. (g) The same location was drilled through, and (h) the corresponding ERT result is shown.

again, used both undamaged and damaged boundary voltage data. The resulting spatial resistivity map is shown in Fig. 9b. One can see from Fig. 9b that the resistivity map successfully identifies localized increases in resistivity corresponding to the location of the partially drilled hole.

The experiment continued by completely drilling through the same location, as can be seen in Fig. 9c. Fig. 9d shows the resistivity map obtained by computing the difference between this new damage state (*i.e.*, through-hole) and the baseline. As

expected, Fig. 9d shows that additional damage accumulated in the structure since the last measurement, and its location is close to that of the actual hole. Similar results were also obtained with the introduction of the next damage state (*i.e.*, partially and completely drilled hole near the top-right corner of the plate), which is shown in Figs. 9e and 9g, respectively. The resistivity maps shown in Figs. 9f and 9h, once again, show the presence of new damage and identify its correct locations and severities. All in all, the results shown in Figs. 8 and 9 demonstrated that there is potential for MWNT-latex thin film-enhanced mortar specimens, when coupled with ERT, to be able to detect spatially distributed structural damage.

Future tests will investigate scalability limits of this technology, especially since tomographic techniques require that electrical current be propagated between all electrodes. High resistivity (or low conductivity) will pose as a severe problem for ERT reconstruction, but a possible solution may be to leverage the unique technique of using MWNT-latex thin films for modifying the interface between cement and sand particles. Other future work will also involve characterizing the electromechanical properties of concrete specimens (*i.e.*, with aggregates) enhanced with these MWNT-latex thin films.

V. CONCLUSIONS

In this study, a novel technique of incorporating carbon nanotubes in cementitious composites was proposed, which is by modifying the cement-sand particle interface using highly conductive, percolated CNT-based thin films. In particular, an airbrushing technique was employed for depositing MWNT-latex thin films onto sand before being used for casting mortar specimens. Then, untreated and film-coated sand were used for casting $5 \times 5 \times 5 \text{ cm}^3$ cubic and $7 \times 7 \times 1 \text{ cm}^3$ plate specimens. The mechanical, nominal electrical, and electromechanical properties of both pristine and MWNT-based mortar specimens were characterized and compared.

First, monotonic, uniaxial compressive load tests revealed the stress-strain characteristics of these mortar specimens. It was found that the Young's moduli of pristine and MWNT-based mortar were 14 and 6 GPa, respectively. The differences were statistically significant in that the MWNT-latex coating on the sand appeared to have modified the mortar's intrinsic mechanical properties. Similar degradations were also observed for compressive strength results.

Second, the electrical and sensing characterization tests showed that incorporation of MWNT-latex thin films decreased their resistivity by more than an order of magnitude. In addition, while both types of mortar specimens exhibited electrical properties that were sensitive to strain, those with the films showed bi-linear response to applied strains. At low strains, the sensitivity was extremely high with an average of ~ 229 . At applied strains greater than 0.2%, both types of specimens had comparable sensitivities of ~ 61 to ~ 65 . However, mortar with embedded thin films exhibited greater signal-to-noise ratio, sensing resolution, repeatability, higher conductivity, and less nominal resistance drift.

Lastly, an electrical resistance tomography algorithm was implemented for back-calculating the spatial resistivity map (or distribution) of MWNT-based mortar specimens for damage detection applications. Plate specimens instrumented with 24 boundary electrodes (in a 6×6 pattern) were subjected to artificial damage due to drilled holes at either the center or near the corners of specimens. The results from all the tests showed very promising results, in which the ERT algorithm correctly estimated the spatial resistivity distribution of the mortar plates. In addition, the spatial resistivity maps also showed localized increases in resistivity corresponding to the locations and sizes of drilled holes. These tests successfully validated ERT for spatial damage detection. Future work will consider the use of MWNT-latex films for modifying cement-sand-aggregate interfaces, as well as how to scale up the technology for real-world and full-scale applications.

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